Attorney Docket: 132/42381CO
PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants: Lawrence Robert GRZYLL et al.

Serial No.: 08/895,687 Group Art Unit 1208

Filed: July 17, 1997 Examiner: Joseph D. Anthony

Title: FIRE EXTINGUISHING METHODS AND BLENDS UTILIZING UNSATURATED PERFLUOROCARBONS

DECLARATION UNDER 37 CFR 1.132

Assistant Commissioner for Patents
Washington, D.C. 20231

Sir:

DECLARATION UNDER 37 C.F.R. §1.132

I, LAWRENCE ROBERT GRZYLL, hereby declare that:

1. I am a co-inventor of the invention disclosed and claimed in the above-referenced U.S. patent application.

2. I received a Bachelor of Science in Chemical Engineering (with high honors) and a Master of Science in Chemical Engineering from the Florida Institute of Technology. I am currently a Ph.D. candidate in Chemical Engineering at the Florida Institute of Technology.

3. I have been employed as a Senior Chemical Engineer at Mainstream Engineering Corporation since 1987.

4. I am familiar with and understand the prosecution history of the above-referenced U.S. pat nt application, as well as the prior art cited by the Examiner; in particular Japanese laid open (kokai) patent application No. Hei 05-42230 ("JP '230"), Pitts et

al., "Construction of an Exploratory List of Chemicals to Initiate the Search for Halon Alternatives" (August 1990) ("Pitts"), and U.S. Patent No. 5,117,917, to Robin et al. ("Robin").

5. In my previous Declaration, signed on January 7, 1997, which is incorporated herein by reference ("my first Declaration"), I established that none of the compounds disclosed in JP '230 are as effective as the common halons. More specifically, none of the compounds disclosed in Table 1 of JP '230 has a flame extinguishing concentration comparable with the common halons. For example, $CF_3CF=CF_2$ in Table 1 has a flame extinguishing concentration of 7.9 vol% in a n-heptane cup burner test. The common halons, such as halon 1301 and 1211, have substantially lower flame extinguishing concentrations of 3.1% and 3.4 % (v/v) respectively.

6. In my first Declaration I established that the composition used in the method of the present invention advantageously extinguishes fires as effectively as the common halons, but without the environmental hazards of the latter. Flame extinguishing compositions consisting of octafluoro-2-butene were found to be effective fire extinguishing compositions at flame extinguishing concentrations for n-heptane diffusion flames in a cup burner test of at most about 5.2% (v/v). I also established that fire extinguishing compositions of octafluoro-2-butene and a mixture of conventional fire extinguishing agents were also found to be effective fire extinguishing compositions at concentrations similar to those of the common halons. In fact, we did not expect at the outset, but were surprised to find that, the flame

extinguishing concentration of compositions consisting of octafluoro-2-butene and a mixture of conventional fire xtinguishing agents were lower than the flame extinguishing concentrations of the individual agents of the composition, depending on the amount of each agent present.

7. In the Office Action dated January 24, 1997, the Examiner commented on my first Declaration saying it "does not use the closest prior-art which is deemed to be the above applied prior-art. It is held that the JP's fluorinated unsaturated hydrocarbons are the closest prior art as opposed to the fluorinated saturated hydrocarbons used in applicants' comparison test."

8. JP '230 itself teaches that the two fluorinated unsaturated hydrocarbons disclosed in the reference, hexafluoroisobutene (compound (8)) and hexafluoropropene (compound (18)) exhibit cup burner flame extinguishing concentrations of 6.7% and 7.9%, respectively.

9. Octafluoro-2-butene, as discussed in the instant application, exhibits a cup burner flame extinguishing concentration of 4.0%. Various runs described in my first Declaration, showed concentrations ranging from 4.0% to 5.1%, averaging 4.7%.

10. The halons have cup burner flame extinguishing concentrations of 3.0% to 3.5%.

11. I conclude from these data that the cup burner flame extinguishing concentration of octafluoro-2-butene (4.0 - 5.1%) is un xpectedly and surprisingly lower than the cup burner flame

extinguishing concentrations of the hexafluoroisobutene and hexafluoropropene of JP '230 (6.7% and 7.9, respectively), and closer to that of the halons.

12. The published literature suggests that the two compounds of JP '230 cited by the Examiner may be toxic and thus not suitable for fire suppression. Sax and Lewis assign a hazard rating of 3 to hexafluoropropene compared to a hazard rating of 1 for octafluoro-2-butene. The 4-hour LC_{50} (concentration that causes deaths in 50 % of the subjects) for hexafluoropropene is 750 ppm (mouse) and 1673 (rat). The 4-hour LC_{50} for hexafluoroisobutene is 1425 ppm (rat). The LC_{10} (the lowest concentration to cause a death in a single subject) for octafluoro-2-butene is 6100 ppm (rat). The LC_{50} is higher than the LC_{10} by definition. Thus, hexafluoropropene is at *least* 3.65 times more toxic than octafluoro-2-butene, and hexafluoroisobutene is at *least* 4.28 times more toxic than octafluoro-2-butene .

13. In the Office Action dated January 24, 1997, the Examiner analyzed the data in my first Declaration, asserting that the difference in the range of 4.0% to 5.1% is 27.5%. From this calculation, the Examiner drew the conclusion that 27.5% variations can occur in cup burner flame extinguishing concentration measurements. He then calculated that the cup burner flame extinguishing concentrations of hexafluoropropene and hexafluoroisobutene could reach as low as 5.73% and 4.86%, respectively. The Examiner's assumptions and methodology are flawed.

14. The Examiner did not correctly apply standard experimental error analysis methods to the data. There are several ways to treat errors in experimental data; one can calculate the average error of the measurements or one could calculate the standard deviation of the measurements. Below is a treatment of the data in my first Declaration:

Data Values: 4.0, 4.0, 4.9, 4.7, 5.0, 4.9, 5.0, 5.1

Average Value: 4.7

Errors From Average (absolute value of avg. minus data):
0.7, 0.7, 0.2, 0.0, 0.3, 0.2, 0.3, 0.4

Average Error: 0.35

Standard Deviation: 0.4472

$$s.d. = \sqrt{\frac{1}{N-1}\sum (x_i - x_{avg})^2}$$

Of these standard error treatments, the largest is the standard deviation. Therefore, one could conclude that my first Declaration established that the flame extinguishing concentration of octafluoro-2-butene is 4.7 ± 0.45. This standard deviation is 9.57% of the average value.

15. If one were to assume that variations of similar magnitude occur for other fire extinguishing agents when tested in a n-heptane diffusion cup burner test, then the flame extinguishing concentration for hexafluoropropene as taught in JP '230 could range as low as 7.14% (7.9% x 0.9043 = 7.14%). Similarly, th flame extinguishing concentration for hexafluoroisobutene could

range as low as 6.06% (6.7% x 0.9043 = 6.06%). These values certainly are not in the range of the claims of our patent application.

16. Another factor militating against choosing the compounds of JP '230 is their availability. We reviewed the catalogs of four manufacturers of fluorinated research chemicals: Oakwood Research Chemicals (West Columbia, South Carolina), PCR, Inc. (Gainesville, Florida), SynQuest Labs, Inc. (Alachua, Florida), and Fluorochem, Ltd. (United Kingdom) for hexafluoroisobutene. This chemical was not in any of their catalogs, indicating that the manufacturability of this compound is either too expensive or not practical to offer as a product.

I hereby declare that all statements made of my own knowledge in the foregoing Declaration are true and that all statements on information and belief are believed to be true, and further that these statements are made with the knowledge that willful false statements and the like are punishable by fine or imprisonment, or both, under § 101 of Title 18 of the United States Code, and may jeopardize the application or this document or any patent issuing thereon.

Date: February 10, 1998

Lawrence Robert GRZYLL

Attorney Docket: 132/42381CO
PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: LAWRENCE ROBERT GRZYLL ET AL.

Serial No.: 08/895,687 Group Art Unit: 1721

Filed: JULY 17, 1997 Examiner: J. ANTHONY

Title: FIRE EXTINGUISHING METHODS AND BLENDS UTILIZING UNSATURATED PERFLUOROCARBONS

DECLARATION UNDER 37 C.F.R. §1.132

Assistant Commissioner for Patents
Washington, D.C. 20231

Sir:

I, LAWRENCE ROBERT GRZYLL, hereby declare that:

1. I am a co-inventor of the above-identified U.S. patent application.

2. I received a Bachelor of Science in Chemical Engineering (with high honors) and a Master of Science in Chemical Engineering from the Florida Institute of Technology.

3. I have been employed as a Senior Chemical Engineer at Mainstream Engineering Corporation since 1987.

4. I am familiar with and understand the prosecution of the above-identified U.S. patent application, as well as the following references cited by the Examiner: Japanese Laid-Open (Kokai) Patent Application No. Hei 05-42230 ("JP '230"); Pitts et al., "Construction of an Exploratory List of Chemicals to Initiate the Search for Halon Alternatives" (August 1990) ("Pitts"); and U.S. Patent No. 5,117,917 to Robin et al. ("Robin").

5. The following tests were conducted under my supervision and/or control:

Agent	Cup Burner FEC (vol%)	Minimum Application Density (lb/ft^2-min)
perfluoro-2-butyltetrahydrofuran	3.5	No Extinguishment
heptafluoropropyl-1,2,2,2-tetrafluoroethyl ether	4.3	3.11
octafluoro-2-butene	4.9	2.28-2.58
octafluoro-2-butene/1-bromopropane (92:8)	No Data	1.47

6. The cup burner flame-extinguishing concentration (FEC) is the concentration of agent that extinguishes a flame as disclosed in the specification at pages 4-5. FEC values are used in screening and ranking agents for total flooding applications. Flooding applications are those in which an entire room is filled with the agent. For streaming applications, the minimum application density (flow rate/fire surface area) is used to rank agents. Streaming applications are those in which the agent is applied directly to a fire in the form of a stream, as with a portable fire extinguisher.

7. Based on the cup burner FEC values in the above Table, one would expect that perfluoro-2-butyltetrahydrofuran would be the most effective fire extinguishing agent, followed by heptafluoropropyl-1,2,2,2-tetrafluoroethyl ether, with octafluoro-2-butene performing the worst.

Surprisingly and unexpectedly, the above Table shows that the ranking of the agents for streaming applications was opposite to what would have been expected to one of ordinary skill in the art based upon the FEC's for total flooding applications.

8. I hereby declare that all statements made of my own knowledge in the foregoing declaration are true and that all statements on information and belief are believed to be true, and further that these statements are made with the knowledge that willful false statements and the like are punishable by fine or imprisonment, or both, under §101 of Title 18 of the United States Code, and I jeopardize the application or this document or patent issuing thereon.

Date: August 27, 1998

8/27/98
Lawrence Robert GRZYLL

Attorney Docket: 132/42381CO
PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant. LAWRENCE ROBERT GRZYLL ET AL.

Serial No.: 08/895,687 Group Art Unit: 1721

Filed: JULY 17, 1997 Examiner: J. ANTHONY

Title: FIRE EXTINGUISHING METHODS AND BLENDS UTILIZING UNSATURATED PERFLUOROCARBONS

DECLARATION UNDER 37 C.F.R. §1.132

Assistant Commissioner for Patents
Washington, D.C. 20231

Sir:

I, LAWRENCE ROBERT GRZYLL, hereby declare that:

1. The following cup burner flame concentration tests were conducted under my supervision and/or control:

Hexafluoropropene % (v/v)	Octafluoro-2-butene % (v/v)
5.5	4.0
5.2	4.0
6.0	4.9
6.8	4.7
6.1	5.0
6.1	4.9
6.4	5.0
6.2	5.1
6.3	
6.1	
Average 6.1	Averag 4.7
Std Dev. 0.447338	Std. Dev. 0.447214

2. The cup burner flame-extinguishing concentration (FEC) is the concentration of agent that extinguishes a flame as disclosed in the specification at pages 4-5. As shown in the above Table, the average FEC value for hexafluoropropene does not fall within the standard deviation from the average FEC value for octafluoro-2-butene.

3. I hereby declare that all statements made of my own knowledge in the foregoing declaration are true and that all statements on information and belief are believed to be true, and further that these statements are made with the knowledge that willful false statements and the like are punishable by fine or imprisonment, or both, under §101 of Title 18 of the United States Code, and I jeopardize the application or this document or patent issuing thereon.

Date: 11/2/98

Lawrence Robert GRZYLL